

UNITED STATES PATENT APPLICATION FOR:

METHOD AND APPARATUS FOR INFILM DEFECT REDUCTION FOR  
ELECTROCHEMICAL COPPER DEPOSITION

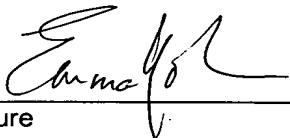
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## **METHOD AND APPARATUS FOR INFILM DEFECT REDUCTION FOR ELECTROCHEMICAL COPPER DEPOSITION**

### **BACKGROUND OF THE INVENTION**

#### **Field of the Invention**

[0001] Embodiments of the invention are generally related to a method for minimizing defects resulting from thermal shock encountered by a substrate during transfer from a fluid processing cell into an annealing chamber.

#### **Description of the Related Art**

[0002] Metallization of sub-quarter micron sized features is a foundational technology for present and future generations of integrated circuit manufacturing processes. More particularly, in devices such as ultra large scale integration-type devices, *i.e.*, devices having integrated circuits with more than a million logic gates, the multilevel interconnects that lie at the heart of these devices are generally formed by filling high aspect ratio, *i.e.*, greater than about 4:1, interconnect features with a conductive material. The most common conductive material used in large scale integration devices is copper. Copper is generally deposited into the high aspect ratio features of these devices using plating processes, such as electrochemical plating (ECP) and/or electroless plating.

[0003] In an ECP process, for example, high aspect ratio features formed into the surface of a substrate, which generally have a conductive seed layer deposited thereon, are filled with a conductive material. ECP processes are generally performed in a two stages. First, the seed layer is formed over the surface features, generally through PVD, CVD, or other deposition process. Second, the surface features of the substrate having the seed layer thereon are exposed to an electrolyte solution, while an electrical bias is applied between the seed layer and an anode positioned in the solution. The solution contains the conductive material to be plated onto the surface of the substrate, and the application of the electrical bias between the seed layer and the anode is configured to cause the conductive material in the

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solution to be plated onto the seed layer and into the interconnect features, thus filling the features.

[0004] Once the plating process is completed, the substrate is generally transferred to at least one of a substrate rinsing cell or a bevel edge clean cell. Bevel edge clean cells, often called IBC cells, are generally configured to dispense an etchant onto the perimeter of the substrate to remove unwanted metal plated thereon. The substrate rinse cells, often called spin rinse dry cells, or "SRD" cells, generally operate to rinse the entire surface of the substrate, front and/or back, with a rinsing and/or cleaning solution to remove any excess processing fluids or contaminants therefrom. The SRD cells are also generally configured to spin the substrate at a high rate of speed in order to spin off any fluid droplets adhering to the substrate surface. Once the remaining fluid droplets are spun off, the substrate is generally clean and dry.

[0005] Once the substrate is clean and dry, the substrate is generally exposed to an increased temperature to stabilize film properties, such as the crystalline structure and the resistivity of the film. For this portion of the process, the substrate may be transferred to an annealing station. A typical annealing station may include an enclosure having a heated substrate support member positioned therein. Alternatively, a substrate support member may be used to support the substrate, while a heating source, such as heating lamps, is used to heat the substrate. Regardless of the heating source used, the annealing station is generally configured to increase the temperature of the substrate from room temperature to between about 200°C and about 400°C in less than about 1 minute.

[0006] However, one challenge with conventional plating systems is that the rapid increase in the temperature of the substrate in the annealing chamber has been shown to cause voids and cracking in the plated layer and between the plated layer and the adjoining dielectric layer. Voids and cracks in the plated layer may be reduced by slowing the anneal temperature ramp, however, slowing the temperature

ramp inherently slows the throughput of the ECP process, which is critical to semiconductor processing.

[0007] Therefore, there is a need for an ECP system and method for processing substrates, wherein the system and method includes an annealing step that both maximizes throughput and minimizes voids and cracking that result from rapid temperature ramp processes.

### **SUMMARY OF THE INVENTION**

[0008] Embodiments of the invention generally provide a semiconductor processing apparatus and method configured to minimize voids and cracks in films resulting from rapid anneal temperature ramping. The apparatus of the invention includes a fluid processing cell configured to preheat the substrate prior to the substrate being transferred to the annealing chamber. The fluid processing cell that is used to preheat the substrate is generally an SRD cell. The method of the invention generally includes supplying a heated fluid to an SRD cell on a semiconductor processing platform. The heated fluid is used to increase the temperature of the substrate to a temperature between room temperature and the annealing temperature prior to the substrate being transferred to the annealing chamber. Further, the heated fluid is applied as part of a previously required processing step, *i.e.*, a rinsing step, and as such, the application of the heated fluid does not have a negative impact on the throughput of the system. Preheating prior to anneal may also be used to shorten the required anneal time, and as such, increase throughput of the processing system.

[0009] Embodiments of the invention may further provide a method for processing a substrate. The method includes plating a conductive layer onto a substrate, transferring the substrate from a plating cell to a cleaning cell, cleaning the substrate in the cleaning cell via application of a heated cleaning fluid to the substrate, drying the substrate in the cleaning cell, transferring the substrate from the cleaning cell to an annealing chamber, and annealing the substrate in the annealing chamber at a temperature of between about 150°C and about 450°C.

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[0010] Embodiments of the invention may further provide a method for processing a substrate, wherein the method includes plating a conductive layer onto a substrate, rinsing the substrate of unwanted residue chemicals, preheating the substrate during the rinsing process to a temperature of between about 25°C and about 100°C, and annealing the substrate in an annealing chamber at a temperature of up to about 450°C subsequent to the preheating process.

[0011] Embodiments of the invention may further provide an apparatus for processing a substrate, wherein the apparatus includes a plating cell positioned on a processing platform, the plating cell being configured to plate a conductive layer onto the substrate, a rinsing cell positioned on the processing platform, and an annealing station positioned on the processing platform. The rinsing cell generally includes a substrate support member configured to support the substrate for processing, a fluid dispensing nozzle positioned to dispense a rinsing solution onto the substrate, and a fluid heating assembly positioned in fluid communication with the fluid dispensing nozzle, the fluid heating assembly being configured to supply a heated rinsing solution at a temperature of between about 50°C and about 100°C.

**BRIEF DESCRIPTION OF THE DRAWINGS**

[0012] So that the manner in which the above recited features of the present invention can be understood in detail, a more particular description of the invention, briefly summarized above, may be had by reference to embodiments, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only typical embodiments of this invention and are therefore not to be considered limiting of its scope, for the invention may admit to other equally effective embodiments.

[0013] Figure 1 illustrates a top plan view of one embodiment of an electrochemical plating system of the invention.

[0014] Figure 2 illustrates an exemplary embodiment of a plating cell used in the electrochemical processing system of the invention.

[0015] Figure 3 illustrates a partial perspective and sectional view of an exemplary substrate spin rinse dry cell of the invention.

[0016] Figure 4 illustrates a partial perspective and sectional view of an exemplary substrate rinse and dry cell of the invention.

#### **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT**

[0017] Embodiments of the invention generally provide an electrochemical plating system configured to plate conductive materials onto semiconductor substrates. The plating system generally includes a substrate loading area in communication with a substrate processing platform. The loading area is generally configured to receive substrate containing cassettes and transfer substrates received from the cassettes into the plating system for processing. The loading area generally includes a robot configured to transfer substrates to and from the cassettes and to the processing platform or a substrate annealing chamber positioned in communication with the loading area. The processing platform generally includes at least one substrate transfer robot and a plurality of substrate processing cells, *i.e.*, ECP cells, bevel clean cells, spin rinse dry cells, substrate cleaning cells, and/or electroless plating cells.

[0018] Figure 1 illustrates a top plan view of an exemplary ECP system 100 of the invention. ECP system 100 includes a factory interface 130, which is also generally referred to as a substrate loading station. The factory interface 130 includes a plurality of substrate loading locations (not shown) configured to interface with substrate containing cassettes 134. A robot 132 is positioned in the factory interface 130, and is configured to access the substrates contained in cassettes 134. Further, robot 132 also extends into a link tunnel 115 that connects the factory interface 130 to a substrate processing mainframe or platform 113. The factory interface robot 132 generally includes the ability to rotate, extend, and vertically move an attached substrate support blade, while also allowing for linear travel along a robot track that generally extends from the factory interface 130 to the mainframe 113.

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[0019] The position of the robot 132 allows the robot 132 to access substrate cassettes 134 positioned on the loading stations, and to then deliver the substrates to one of the processing cell stations shown at 114 and 116 on the mainframe 113. Similarly, the robot 132 may be used to retrieve substrates from the processing cells 114, 116, or transfer substrates to or from an annealing chamber 135. After a substrate processing sequence is complete, robot 132 generally operates to return substrates to one of the cassettes 134 for removal from the ECP system 100. Additional configurations and implementations of an electrochemical processing system are illustrated in commonly assigned United States Patent Application Serial No. 10/435,121 filed on December 19, 2002 entitled "Multi-Chemistry Electrochemical Processing System", which is incorporated herein by reference in its entirety.

[0020] The anneal chamber 135 generally includes a two position annealing station, wherein a cooling plate 136 and a heating plate 137 are positioned adjacently with a substrate transfer robot 140 positioned proximate thereto, e.g., between the two plates. The robot 140 is generally configured to move substrates between the respective heating 137 and cooling plates 136. Further, although the anneal station 135 is illustrated as being positioned such that it is accessed from the link tunnel 115, embodiments of the invention are not limited to any particular configuration or placement. As such, the anneal station 135 may be positioned in communication with the mainframe 113. Additional information relative to the anneal station 135 of the invention may be found in a commonly assigned U.S. Patent Application Serial No. 60/463,860, entitled "Two Position Anneal Chamber," which is hereby incorporated by reference in its entirety.

[0021] ECP system 100 also includes a processing mainframe 113. A substrate transfer robot 120 is positioned on the mainframe 113, and includes one or more blades 122, 124 configured to support and transfer substrates. Additionally, robot 120 and the accompanying blades 122, 124 are generally configured to extend, rotate about a central point, and vertically move, so that the robot 120 may insert and remove substrates from a plurality of processing cells 102, 104, 106, 108, 110,

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112, 114, 116 positioned on the mainframe 113. Generally, processing cells 102, 104, 106, 108, 110, 112, 114, 116 may be any number of processing cells utilized in an electrochemical plating process, e.g., electrochemical plating cells, rinsing cells, bevel clean cells, spin rinse dry cells, substrate surface cleaning cells, electroless plating cells, metrology inspection stations, and/or other processing cells that may be beneficially used in conjunction with a plating process. Each of the respective processing stations and robots are generally in communication with a process controller 111, which may be a microprocessor-based control system configured to receive inputs from a user and/or various sensors positioned on the system 100 and appropriately control the operation of the system 100 in accordance with the inputs and/or a predetermined control sequence.

[0022] In the exemplary plating system illustrated in Figure 1, processing stations 114 and 116 may be configured as an interface between the wet processing stations on mainframe 113, e.g., plating cells, cleaning cells, etc., and the dry processing regions in the link tunnel 115, annealing chamber 135, and the factory interface 130. The processing cells located at the interface stations may be spin rinse dry cells or other type of substrate cleaning cells. More particularly, processing cells 114 and 116 may include both a spin rinse dry cell and/or a substrate cleaning cell in a stacked configuration. Processing cells 102, 104, 110, and 112 may be configured as plating cells, such as electrochemical plating cells or electroless plating cells. Processing cells 106, 108 may be configured as substrate bevel cleaning cells, which are also referred to as IBC cells. Additional configurations and implementations of an electrochemical processing system are illustrated in commonly assigned United States Patent Application Serial No. 10/435,121 filed on December 19, 2002 entitled "Multi-Chemistry Electrochemical Processing System," which is incorporated herein by reference in its entirety.

[0023] Figure 2 illustrates a partial perspective and sectional view of an exemplary electrochemical plating cell 200 that may be implemented in processing cell locations 102, 104, 110, and 112. The electrochemical plating cell 200 includes an outer basin 201 and an inner basin 202 positioned within outer basin 201. The



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inner basin 202 is generally configured to contain a plating solution that is used to plate a metal, e.g., copper, onto a substrate during an electrochemical plating process. During the plating process, the plating solution is generally continuously supplied to inner basin 202, and as such, the solution continually overflows the uppermost point (generally termed a "weir") of the inner basin 202, and is collected by an outer basin 201. The plating solution is then drained and collected for chemical management and/or recirculation. The frame member 203 of plating cell 200 supports an annular base member on an upper portion thereof. Since frame member 203 is elevated on one side, the upper surface of base member 204 is generally tilted from horizontal at an angle that corresponds to the angle of frame member 203 relative to a horizontal position. Base member 204 includes an annular or disk shaped recess formed into a central portion thereof, the annular recess being configured to receive a disk shaped anode member 205. Base member 204 further includes a plurality of fluid inlets/drains 209 extending from a lower surface thereof. Each of the fluid inlets/drains 209 are generally configured to individually supply or drain a fluid to or from either the anode compartment or the cathode compartment of plating cell 200. Anode member 205 generally includes a plurality of slots 207 formed therethrough, wherein the slots 207 are generally positioned in parallel orientation with each other across the surface of the anode 205. The parallel orientation allows for dense fluids generated at the anode surface to flow downwardly across the anode surface and into one of the slots 207. Plating cell 200 further includes a membrane support assembly 206. Membrane support assembly 206 is generally secured at an outer periphery thereof to the base member 204, and includes an interior region configured to allow fluids to pass therethrough.

[0024] A membrane 208 is stretched across the support 206. The membrane operates to fluidly separate catholyte chamber and anolyte chamber portions of the plating cell 200. The membrane support assembly may include an o-ring type seal positioned near a perimeter of the membrane 208, wherein the seal is configured to prevent fluids from traveling from one side of the membrane secured on the membrane support 206 to the other side of the membrane 208. A diffusion plate 210, which is generally a porous ceramic disk member, is configured to generate a

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substantially laminar flow or even flow of fluid in the direction of the substrate being plated. The diffusion plate 210 is positioned in the cell 200 between membrane 208 and the substrate being plated. The exemplary plating cell is further illustrated in commonly assigned United States Patent Application Serial No. 10/268,284, which was filed on October 9, 2002 under the title "Electrochemical Processing Cell", claiming priority to United States Provisional Application Serial No. 60/398,345, which was filed on July 24, 2002, both of which are incorporated herein by reference in their entireties.

[0025] Figure 3 illustrates a partial perspective and sectional view of an exemplary substrate spin rinse dry cell 300 of the invention. The spin rinse dry cell 300 (SRD) includes a fluid bowl 301 supported on a frame that may be attached to a plating system, such as the mainframe 113 illustrated in Figure 1. The SRD 300 further includes a rotatable flywheel 302 centrally positioned in the fluid bowl 301. The flywheel 302 may include a generally planar or curved upper surface that has a plurality of backside fluid dispensing nozzles 308 formed thereon and at least one gas dispensing nozzle 310 formed thereon. These nozzles 308, 310 permit fluid, e.g., deionized water, a cleaning solution, and/or gas, e.g., N<sub>2</sub> purge gas, to be applied to the backside of a substrate 304. In one embodiment, flywheel 302 is covered by a horizontal shield 330 on an upper surface thereof, and by a vertical shield 331 on a side or vertical surface thereof. Both shields 330, 331 are positioned to be stationary and adjacent to the flywheel 302. More particularly, horizontal shield 330 may be attached to the central hub 320 and extend radially outward therefrom. Further, shield 330 may be positioned to essentially float above the rotating flywheel 302 with a space between the rotating flywheel 302 and the shield 330 being between about 1mm and about 5mm, for example. Similarly, vertical shield 331 may be attached to basin shield member 312 and be positioned to be spaced from a vertical edge of the flywheel 302 by a distance of between about 1mm and about 5mm, for example. The positioning of shields 330, 331 is generally configured to minimize the exposed rotating surface area of flywheel 302. More particularly, the exposed surface area 332 of flywheel 302 is known to cause turbulent airflow in cell 300. Since turbulent airflow does not facilitate effective

drying of substrates, minimization of turbulent airflow is desired. Thus, in one embodiment of the invention, the exposed rotating surface area of the flywheel 332 is minimized in order to minimize induced turbulence in the airflow within the cell 300.

[0026] A plurality of upstanding substrate engaging fingers 303 are positioned radially around the perimeter of flywheel 302. Generally, fingers 303 are airfoil shaped when viewed from the top, so that the fingers 303 will generate minimal turbulence when flywheel 302 is rotated. In the illustrated embodiment of the invention, four fingers 303 may be utilized, however, the invention is not limited to any particular number of fingers. Fingers 303 are configured to rotatably support a substrate 304 at the bevel edge thereof for processing in SRD 300. Together, the flywheel 302 and the substrate engaging fingers 303 serve as a rotatable substrate support member. However, other embodiments may be provided where the engaging fingers 303 are connected to the side wall or other components of the cell than a flywheel.

[0027] The processing cell 300 also includes a fluid dispensing arm 350 that may be pivotally mounted to the side wall, or a structure positioned outside of the cell 300, such that a distal end of the arm having a fluid dispensing nozzle positioned thereon may be pivoted to a position over a substrate 304 being processed in the cell 300. The pivotal motion of the arm 350 is generally in a plane that is parallel and above the substrate 304 being processed. The pivotal movement of the arm 350 allows the nozzle positioned on the end of the arm 350 to be positioned over specific radial positions on the substrate, *i.e.*, over the center of the substrate or over a point that is a specific radius from the center of the substrate 304, for example.

[0028] Processing cell 300 also includes an upper cell wall 309 attached to the catch cup 314 and curved surface 316, all of which may be raised and lowered to facilitate loading and unloading of substrates. For example, when a substrate is loaded, upper wall 309 may be raised from cell bowl 301 to allow for access to the substrate engaging fingers 303. When processing begins, then wall 309 may be

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lowered to position the catch cup 314 and curved wall 316 next to the substrate so the that the fluid spun off of the substrate may be captured and airflow over the perimeter of the substrate controlled. Exemplary processing cells that may be used to advantage to practice the invention include commonly assigned United States Patent Application Serial No. 10/680,616, filed October 6, 2003 and 6,290,865, both of which are hereby incorporated by reference in their entireties.

[0029] Processing cell 300 also includes a heating source configured to increase the temperature of the substrate during a fluid processing step. The heating source may include a heated fluid source 375 in fluid communication with the fluid dispensing arm 350 and/or the backside nozzles 308. The source of heated fluid 375 may include a fluid tank having a resistive heating element 382 positioned therein. Heating element 380 is in electrical communication with a source of power 382. The source of power 382 may be in communication with controller 111, and as such, be controlled by controller 111 illustrated in Figure 1. The tank may be in fluid communication with another tank or supply, such as a deionized water supply, that is configured to supply fresh rinsing fluid to the tank. Other fluid sources that may be in communication with the tank include cleaning solutions, etching solutions, and/or other solutions that may be useful in an electrochemical plating process, such as acids, peroxides, and mixtures thereof. One exemplary solution may be a combination of an acid and peroxide, which is generally used to conduct a bevel etching process in semiconductor processing. Further, the source of heated fluid 375 may include a fluid temperature measuring device, e.g., a thermocouple 381, positioned in the tank to measure the temperature of the fluid in the tank. The thermocouple 381 may be in electrical communication with the system controller 111, and as such, the system controller 111 may be used to control the temperature of the fluid in the tank by controlling the operation of the heating element 380 in accordance with data received from the thermocouple 381 and/or a processing recipe. Therefore, the source of heated fluid 375 may be generally configured to maintain a fluid solution therein at a predetermined temperature.

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[0030] In another embodiment of the invention, the source of heated fluid 375 may be replaced or supplemented with heating lamps 402 positioned to heat the substrate during the fluid processing step, as illustrated on processing cell 400 in Figure 4. In this configuration, the heating lamps 402 may be used to apply radiant heat to the substrate while the rinsing solution is being applied to the substrate, which increases the substrate temperature during a rinsing step without having to provide a heated fluid. Further, the heating lamp may remain on during the spin dry process, which operates to further increase the substrate temperature even after the rinsing step is completed. Additionally, in this configuration, a temperature monitoring apparatus may be used to monitor the temperature of the substrate and to control the application of electrical power to the lamps 402 in accordance with the monitored temperature and a control program, for example. In addition to conducting the pre anneal-type process described above, the heated fluid and/or the heating lamps 402 may be used to completely anneal a substrate. For example, process cell 300, 400 may be used to rinse, dry, and anneal a substrate. In this embodiment the processing time in the cell may be increased so that the temperature of the substrate may be increased to between about 100° C and about 450° C via application of the heated fluid and/or radiant heat from the lamps 402. The duration of the annealing process in cells 300, 400 may be between about 30 seconds and about 3 minutes, or between about 30 seconds and 90 seconds, for example. Other than the addition of heating lamps 402, processing cell 400 is generally similar to processing cell 300, and as such, numbering has been preserved in the respective figures. However, in the embodiment where cell 400 is used for a complete annealing process, cell 400 may be modified to include an enclosure that is configured to isolate the processing cell environment from ambient. Additionally, the processing cell environment may be in fluid communication with a vacuum pump and/or a process gas supply, such as nitrogen, hydrogen, helium, argon, etc. The combination of the vacuum pump and gas supply allows for the processing environment of the cell to be controlled, and more particularly, for the oxygen content to be minimized.

[0031] In operation, embodiments of the invention are generally configured to preheat a substrate prior to an anneal process in order to minimize voids, cracks, and peeling associated with the thermal shock of placing a room temperature substrate into a high temperature annealing chamber. The preheating process is generally conducted in a fluid processing cell, and more particularly, in an SRD cell positioned on the processing platform, wherein the SRD cell is configured to dispense a heated fluid onto the substrate to increase the temperature of the substrate prior to transferring the substrate to the anneal chamber.

[0032] As noted above, a conventional ECP process includes plating a conductive layer onto a substrate, transferring to a bevel edge cleaning cell for bevel cleaning, transferring to a spin rinse dry cell for rinsing and/or cleaning and drying the substrate, and then transferring to an anneal chamber where the substrate is heated to stabilize the conductive layer. The present invention adds a substrate heating step into the spin rinse dry process. More particularly, a conventional substrate spin rinse dry process includes rotating the substrate at a rate of between about 10 rpm and about 500 rpm while a rinsing and/or cleaning solution is dispensed onto the top and/or bottom surfaces of the substrate. The rinsing fluid of the present invention is provided to the spin rinse dry cell at a temperature of between about 25°C and about 100°C, or alternatively, between about 50°C and about 100°C or between about 75°C and 100°C. The heated rinsing fluid may be applied to the front and/or backside of the substrate while the substrate is rotated. The fluid contacts the substrate surface and transfers heat from the fluid to the substrate, thus heating the substrate to a temperature near the temperature of the rinsing fluid. The heated rinsing fluid may be dispensed onto the substrate until the substrate surfaces are sufficiently clean and the substrate is heated to the desired temperature. In one embodiment of the invention, deionized water at a temperature of between about 50°C and about 100°C is dispensed onto the substrate surface while the substrate is rotated. The heated deionized water is dispensed for between about 5 seconds and about 20 seconds before the flow of the heated water is terminated and a drying process is initiated.

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[0033] Once the rinsing process is completed, the flow of the rinsing fluid is terminated, and the substrate may be rotated at a higher rotation rate to dry the substrate. For example, the substrate may be rotated at between about 500 rpm and about 3000 rpm for between about 10 seconds and about 60 seconds to dry the substrate, or alternatively, between about 5 seconds and about 25 seconds. The higher rotation speed of the substrate generates a centrifugal force sufficient to urge fluid outward and off of the surfaces of the substrate, thus drying the substrate. In the present invention, it is desirable to spin the substrate at between about 2000 rpm and about 3000 rpm during the drying process so that the drying time is minimized, as an extended drying time has been shown to decrease the substrate temperature. Therefore, once the substrate is heated with the heated rinsing fluid, embodiments of the invention are configured to minimize delay and transfer the substrate to the annealing chamber as soon as practicable.

[0034] Once the drying process is completed, the substrate is transferred to the annealing chamber. In the system illustrated in Figure 1, for example, the substrate may be transferred from SRD cell 114, 116 to the annealing chamber 135 by robot 132. Embodiments of the invention contemplate that the timeframe for transfer of the substrate from the SRD cell 114, 116 to the anneal chamber 135 is less than about 30 seconds, and more particularly, less than about 15 seconds. Further, embodiments of the invention contemplate that the timeframe between the time that the heated rinsing fluid is terminated and the time that the substrate is positioned in the anneal chamber 135 may be less than about 90 seconds, or more particularly, between about 20 seconds and about 60 seconds. The transfer time between the rinsing cell and the annealing station is critical to the invention, as longer transfer times allow the substrate to cool between the heated rinsing and the annealing stages of the process, which increases the thermal shock and stress on the substrate. Once the substrate is positioned in the annealing chamber 135, the temperature of the substrate may be increased to between about 150°C and about 450°C, between about 200°C and 400°C, or up to about 450°C.